

TN 2973

NATIONAL ADVISORY COMMITTEE FOR AERONAUTICS

TECHNICAL NOTE 2973

EFFECT OF PRETRAINING ON RECRYSTALLIZATION
TEMPERATURE AND MECHANICAL PROPERTIES
OF COMMERCIAL, SINTERED, WROUGHT
MOLYBDENUM

By Kenneth C. Dike and Roger A. Long

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EFFECT OF PRESTRAINING ON RECRYSTALLIZATION TEMPERATURE

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SUMMARY

An investigation was made of the feasibility of raising the recrystallization temperature of commercial molybdenum by limiting the amount of prestraining without appreciably sacrificing strength or ductility.

The recrystallization temperature of molybdenum decreases with increase in the percent of effective swaging if counteracting variables are absent. The 1-hour total recrystallization temperature of one lot of metal was above 2900° F for 35 percent or less swaging, whereas 99 percent swaging gave a recrystallization temperature of 2300° F. Molybdenum does not always respond to swaging in this manner. Two other lots of metal had an almost constant recrystallization temperature of 2075° F irrespective of swaging. Neither chemical nor X-ray diffraction analyses revealed any significant data which could account for this difference.

The atmosphere used to determine the recrystallization data had a pronounced effect on the 1-hour recrystallization temperature. Metal swaged 50 percent recrystallized at 2300° F in hydrogen, 2700° F in a vacuum, and 2850° F in argon.

Increasing effective swaging caused a small increase in the ultimate tensile strength at room temperature. Metal swaged 10 percent had an average strength of 85,000 pounds per square inch in both the as-swaged and stress-relieved states. The tensile strengths of the 99-percent swaged metal and of the stress-relieved metal averaged 101,000 and 96,000 pounds per square inch, respectively. At 1800° F, tensile strengths varied from 26,000 to 39,000 pounds per square inch, but the difference occurred between lots of metal and not from swaging.

The ductility at room temperature varied within each lot of metal and illustrated that severe prestraining is not necessary to impart

good ductility. However, stress relieving improved the ductility in all cases. At 1800° F, the ductility remained adequate (10 percent or more elongation) with no apparent relation to swaging.

Recrystallized metal, regardless of the amount of prior swaging, possessed mechanical properties inferior to as-swaged or stress-relieved metal tested at room temperature. Although increased swaging produced a considerably finer recrystallized grain size, this was no criterion by which strength or ductility could be predicted for room-temperature tests.

INTRODUCTION

Practical engineering interest in high-purity commercial molybdenum as a high-temperature material has increased tremendously in the last few years and has reached the point where molybdenum is now the object of extensive research and development. Not only are its properties and potential use at elevated temperatures being investigated, but also the possibilities of improving upon these properties by such means as alloying and heat treating are being examined.

The present commercial practice of swaging or rolling ingots of molybdenum into bars and sheets has the effect of imparting a fibrous structure to the metal which possesses superior mechanical properties to those of the ingot or metal subsequently recrystallized (ref. 1). Although swaging increases the strength and ductility, it also has the very detrimental effect of progressively lowering the recrystallization temperature. This limits its use to less than 2000° F and prevents brazing or the application of protective coatings at high temperatures which would recrystallize the metal.

There are two methods whereby the recrystallization temperature of most metals can be influenced. Additions of other elements to a pure metal (impurities in commercial metals may be effective) usually have a tendency to raise the recrystallization temperature. A limited amount of recent data (ref. 2) shows that the recrystallization temperature of arc-cast molybdenum can be raised by the addition of small percentages of other elements. The second method is a fundamental law of recrystallization: the smaller the degree of working (effective strain hardening), the higher the recrystallization temperature. No data to the authors' knowledge are available on the application of this method to molybdenum.

An investigation was initiated at the NACA Lewis laboratory to determine whether molybdenum could be produced, by slight modification of commercial fabrication methods, which would have a higher recrystallization temperature without an appreciable sacrifice in strength or ductility.

The investigation included:

- (1) The effect of swaging on recrystallization temperature.
- (2) The effect of swaging on strength and ductility at room temperature and 1800° F.

Data are also presented on grain size, chemical composition, and X-ray analysis.

MATERIAL PROCESSING

Three 5-kilogram, cold-pressed, sintered molybdenum ingots were obtained from Cleveland Tungsten Inc., over a period of time, and each was swaged by conventional, commercial methods at the time of procurement into 0.225-inch-diameter rods which were designated as lots 1, 2, and 3. The 0.225-inch-diameter rod of each lot was then cut into three or four equal lengths and given additional processing to obtain finished 0.125-inch-diameter rods, each possessing a different amount of induced strain. A master processing flow diagram (fig. 1) illustrates the various steps taken to obtain these finished products. The amounts of induced strain are designated as percentages of swaging, which represent cross-sectional area reductions from fully recrystallized metal. The end products obtained from each lot of material are as follows:

Lot number	Swaging, percent
1	35, 50, 69, 99
2	10, 22, 35
3	10, 35, 50, 99

See figure 2 for photomicrographs of typical structures resulting from the various amounts of swaging.

The swaging procedure for each ingot is shown in table I. The 0.225-inch-diameter rods and all intermediate sizes down to 0.125 inch were examined metallographically, and results indicated that all swaging from 0.225 inch was done below the recrystallization temperature of the material. It is not known if any recrystallization occurred during the swaging at the higher temperatures used to reduce the ingot to the 0.225-inch diameter.

APPARATUS AND PROCEDURE

Short-time tensile evaluation. - Tensile specimens were fabricated with a 1.25 ± 0.02 -inch gage length which was 0.090 ± 0.003 inch in diameter with a ground surface finish of 5 to 16 root mean square units of roughness.

Ultimate-tensile-strength evaluations were made with Templin-type grips and a commercial hydraulic-type tensile machine with a low scale of 6000-pound capacity. Yield strengths were determined with a Templin-type stress-strain recorder. An approximate rate of loading of 300 pounds per minute (47,500 psi) was used for all specimens.

The heating apparatus, instrumentation, and atmosphere protection used to obtain data at 1800° F are described in reference 3.

Recrystallization. - Tensile specimens were recrystallized or stress-relieved either in a dry hydrogen atmosphere with a dew point of -45° F or less or in a vacuum of less than 0.2 micron as detailed in table II.

One-hour recrystallization temperature data were determined for lot 3 in hydrogen and vacuum as previously and also in commercial, tank quality argon. Lots 1 and 2 were evaluated only in argon. Data in hydrogen and argon were obtained by placing specimens in the furnace at the evaluation temperature. Data in vacuum had to be obtained by starting with a cold furnace. Therefore, heating times to temperature were not constant; a temperature of 2000° F required approximately 2.5 hours and 2600° F, about 3.5 hours. Maximum temperature variation in all atmospheres was $\pm 15^{\circ}$ F.

Metallography. - The metallographic techniques used for visual examinations and photomicrographs are detailed in reference 1. Percentages of recrystallization were determined on a longitudinal plane at the center line of 1/2-inch-long specimens. Grain counts of recrystallized metal were made on transverse surfaces by the Jeffries method (ref. 4).

Chemical analyses. - Residual gas analyses were made by a vacuum fusion method and the carbon analyses by conventional methods at Battelle Memorial Institute. The semiquantitative values were determined by the National Spectrographic Laboratories using carbon-arc techniques; the sulphur was determined by conventional chemical methods.

X-ray analyses. - Grain orientation data were obtained with a Geiger counter X-ray diffraction spectrometer. The specimen holder and techniques used are described in reference 5. Back reflection data were procured by standard film techniques and also with a 180° Geiger counter X-ray diffraction spectrometer.

Specimens were ground and polished so that the diffracting surface was a diametral plane parallel to the axis of the swaged bar. Since hand-polishing techniques left a layer of worked metal on the surface, this layer was removed by electropolishing.

RESULTS AND DISCUSSION

Effect of swaging on recrystallization temperature. - Figure 3 shows that the 1-hour, total recrystallization temperature of lot 3 molybdenum in argon was varied over a temperature range of approximately 800° to 1000° F by controlling the amount of final swaging. This swaging effect, however, was not evident in lots 1 and 2. Lot 3 had an increasingly higher recrystallization temperature with decreasing amounts of effective swaging, the 10 and 35 percent swaged material having 1-hour total recrystallization temperatures somewhere above 2900° F. Furnace limitations prevented an exact determination.

Molybdenum with such a high recrystallization temperature has a potential high-temperature use much superior to that of the usual heavily cold-worked metal, which has a much lower recrystallization temperature. Metal with a high recrystallization temperature can be brazed with high-melting-point alloys of superior strength, can be coated with protective materials at higher temperatures, and also can be used at a higher temperature and still retain the superior mechanical properties imparted by working.

Although the recrystallization temperature was raised appreciably for lot 3 by limiting the amount of prestrain, some other unknown variable exists which can exert sufficient influence to completely nullify any effects gained by this method, as shown by lots 1 and 2 in figure 3. These two lots of metal had an almost constant recrystallization temperature, about 2075° F, regardless of the amount of finish swaging, although they were processed in the same way as lot 3 except a 100° to 200° F higher swaging temperature from the 0.5-inch-diameter size to 0.125-inch diameter (table I). This higher finishing temperature would tend to raise and not lower the recrystallization temperature.

Since chemical content can influence the recrystallization temperature, a vacuum fusion analysis (table III) was made on all three lots of metal for the residual gases, oxygen, nitrogen, and hydrogen, which are considered important in molybdenum. Table III also contains conventional chemical analyses for carbon and sulphur and semiquantitative spectrographic analyses for trace elements. None of these analyses showed any significant difference among the three lots which could conceivably cause the difference in properties.

Since it was thought that molybdenum having different recrystallization temperatures might also possess structural differences, an X-ray diffraction study was made of lots 1 and 3 to determine preferred orientation characteristics and residual stress differences. The X-ray diffraction data, obtained with the methods described in reference 5, show that there was no appreciable difference in the degree or type of preferred orientation between lots 1 and 3.

Back-reflection photograms indicate that all specimens of both lots, regardless of the amount of finish swaging, possessed very low residual stress. Figure 4 shows that metal swaged as high as 99 percent produced Debye-Scherrer rings of sharp spots and remarkable resolution of the K_{α} doublet (321), both typical of low residual stress in other metals. Resolution of the K_{α} doublet involves line broadening which was further investigated with a 180° Geiger counter spectrometer. These data also show no difference among lots or the degree of swaging. However, metal from lot 3 (swaged 35 percent) when stress-relieved showed a decrease in line width which indicates the presence of less stress.

Data in reference 6 show that the working temperature is important in duplicating mechanical properties of molybdenum. If partial or total recrystallization occurs at any time during working, a coarse-grained structure is obtained which has inferior mechanical properties. This structure was obtained at 2372° F after a 75-percent reduction in area. Table I shows that the metal used in this investigation was also swaged within $\pm 150^{\circ}$ F of this temperature at 75 percent reduction and it is not known whether any recrystallization occurred during swaging to the 0.225-inch-diameter size. No recrystallization, however, took place during further swaging. It is known that higher working temperatures tend to raise the recrystallization temperature, but no data are available for molybdenum. Since the commercial temperature control was not too good during the initial swaging, it is not known whether the possible temperature differences among lots could have influenced the recrystallization temperature. Although this influence could exist, it is believed unlikely to result in a constant recrystallization temperature irrespective of the amount of final swaging.

The atmosphere used to determine the recrystallization data of lot 3 had a pronounced effect upon the 1-hour recrystallization temperature (fig. 5). This effect is not prominent for heavily swaged (99 percent) material, but can make a tremendous difference (2400° F in H_2 and over 3000° F in A) in metal swaged 35 percent. The slope of the curve obtained in hydrogen is similar to that reported in reference 6 in hydrogen for various amounts of swaging, except that the curve lies approximately 200° F higher. Though lower, the reference data illustrate the change of recrystallization temperature with working, a sharp rise of 300° to 400° F occurring between 33 and 17 percent final working.

A very interesting phenomenon was observed when specimens were recrystallized in argon or in vacuum, but it was never observed when hydrogen was used. At the 1-hour recrystallization temperature of each specimen, all the metal recrystallized except for an irregular surface layer (fig. 6), which averaged 0.005 to 0.015 inch thick. This was

present on both swaged and transverse cut surfaces in all lots of metal with no relation to the amount of swaging. Tests revealed that these edge grains would not recrystallize until held for 1 hour at a temperature 300° to 400° F higher than the 1-hour recrystallization temperatures reported in the data. This phenomenon may result from the diffusion of some gaseous element into or out of the metal. If this could be determined and controlled, it might well be another possibility of producing molybdenum with a high recrystallization temperature.

Effect of swaging on strength and ductility at room temperature and 1800° F. - To be of any practical value, particularly at elevated temperatures, molybdenum given only a light amount of effective swaging must possess mechanical properties not too inferior to heavily swaged material. Figure 7 shows that the ultimate room-temperature tensile strengths of metal given only 10 percent effective prestraining can be within 16 percent that of the average (approximately 101,000 psi) of 99 percent swaged material and also have equivalent ductility. If the metal is swaged 35 percent, the strength differential can be reduced to 5 to 10 percent of the average of the 99 percent swaged metal. This is but a small sacrifice in ultimate tensile strength for a beneficial increase in recrystallization temperature and does not restrict the use of molybdenum at room temperature.

Data in reference 3 show that 50 percent or more swaging is beneficial in lowering the transition temperature of molybdenum and may make the difference between a ductile or brittle metal at room temperature. The ductility curve for lot 3 (fig. 7) shows that for this particular ingot, 35 percent swaging was not sufficient to lower the transition temperature below room temperature. However, there are unknown factors which also have an influence on the transition temperature as shown by lot 2, which was given only 10 to 35 percent final swaging and remained quite ductile.

At elevated temperatures, molybdenum, like other cold-worked metals, is subject to a stress-relieving action which removes residual stresses incurred during fabrication and results in a product with more nearly uniform properties. Figure 7 shows that stress relieving generally lowers the ultimate tensile strength no more than 5 percent, except in the case of the 22 percent swaged metal. It is not known what fabrication variable could have given this particular bar in the as-swaged state an ultimate tensile strength equal to metal swaged 99 percent. However, this high strength is relatively unimportant since it can not be retained at elevated temperatures.

In the stress-relieved condition, molybdenum given only 10 percent effective swaging can have a strength of 86,000 pounds per square inch; the strength increases rather uniformly up to an average value of

96,000 pounds per square inch at 99 percent reduction. This seems a rather slight increase for such a severe reduction. Figure 7 shows stress relieving to be very beneficial in improving the as-swaged ductility at room temperature. Outstanding was the increase of ductility from approximately 6 to 31 percent for the 35 percent swaged metal of lot 3.

The ultimate tensile strength at 1800° F (fig. 8) varied from 26,000 to 39,000 pounds per square inch, the variation depending almost entirely upon the characteristics of each lot and not from any effect of swaging. Within any one lot of metal, the strength differential from swaging is within the experimental scatter band. The curves for lot 2 would probably have been straighter, similar to those for the stress-relieved metal in figure 7, had sufficient time been allowed to effect a complete stress relief while the testing temperature was being attained. The ductility at 1800° F (fig. 8) apparently depends only upon the inherent properties of each ingot and is not affected by various amounts of final swaging. The discontinuity in the ductility curve of lot 2 probably exists for the same reason as mentioned for the strength curve.

Although various amounts of prestraining do not result in large differences in mechanical properties, they do have a tremendous effect on the grain size of metal subsequently recrystallized. A semilogarithmic plot (fig. 9) shows that 99 percent swaged metal recrystallized with a grain size of approximately 1000 to 4000 grains per square millimeter, whereas metal swaged 10 percent had only 7 grains per square millimeter. The differences in grains per square millimeter between lots 1 and 3 are large but differ no more than 2 A.S.T.M. grain-size numbers, which is not considered large in commercial metallurgy. Data in reference 6 on the effect of prestraining on recrystallized grain size of arc-cast molybdenum agree surprisingly well (within 1 A.S.T.M. grain-size number) with the data in this investigation as shown in figure 9.

The inferior and varied mechanical properties of recrystallized metal and also the limited effect of prior swaging are shown in figure 10. A comparison with figure 7 clearly illustrates that as-swaged properties are no criterion by which recrystallized properties can be ascertained. In the as-swaged state, both lots 1 and 3 have equivalent strengths and adequate ductility; however, when recrystallized, lot 1 possesses no ductility at room temperature whereas lot 3 retains adequate ductility. This unpredictable scatter of properties is brought about primarily by an increase in the transition temperature which occurs during recrystallization. It is known (ref. 3) that swaging lowers the transition temperature but this desirable effect is nullified upon subsequent recrystallization. However, some unknown processing or compositional variable exists which exerts sufficient

influence to determine whether the transition temperature shall be above or below room temperature. Although a fine grain size in most metals usually results in superior mechanical properties, it is evident that in recrystallized molybdenum the difference is negligible. The strengths of metal having a grain size of 7 grains per square millimeter were equivalent to those of metal with 1000 to 4000 grains per square millimeter. The elongation of the large-grain-size material was 10 percent, whereas no elongation was obtained for lot 1, with approximately 4000 grains.

SUMMARY OF RESULTS

The following results were obtained from an investigation of the effect of prestraining on the recrystallization temperature and mechanical properties of sintered, wrought molybdenum:

1. The recrystallization temperature of molybdenum varied inversely with the percent of effective swaging when counteracting variables were absent. The 1-hour total recrystallization temperature of one lot of metal was above 2900° F for 35 percent or less swaging, whereas 99 percent swaged metal had a recrystallization temperature of 2300° F. Two other lots of metal had an almost constant recrystallization temperature of approximately 2075° F over the entire working range of 10 to 99 percent. No difference could be detected from either chemical or X-ray diffraction analyses which might account for this variation among lots.

2. The atmosphere used to determine the recrystallization data had a pronounced effect on the 1-hour recrystallization temperature of one lot of molybdenum swaged 50 percent or less. This 50 percent swaged lot recrystallized in 1 hour at 2850° F in argon, at 2700° F in a vacuum, and at 2300° F in hydrogen. In the same atmospheres the recrystallization temperature of 99 percent swaged metal varied only from 2200° to 2300° F.

3. Increasing effective swaging caused a small increase in the ultimate tensile strength at room temperature. The average strength of metal swaged 10 percent was 85,000 pounds per square inch and of metal swaged 99 percent, 101,000 pounds per square inch. When stress-relieved, the 10 percent swaged metal retained the same strength whereas the strength of the 99 percent swaged metal was lowered to 96,000 pounds per square inch. At 1800° F, the ultimate tensile strength varied from 26,000 pounds per square inch to 39,000 pounds per square inch. This difference, however, was not caused by the amount of swaging but by the characteristics of each lot of metal evaluated.

4. The effect of swaging on the ductility of as-swaged molybdenum at room temperature varied with each lot of metal and showed that heavy effective swaging is not necessary to obtain adequate ductility. Stress relieving increased the ductility in all cases and also illustrated that 10 percent swaging can impart ductility equivalent to 99 percent working. At 1800° F, ductility remained adequate (10 or more percent elongation) with no apparent relation to the amount of swaging.

5. Recrystallized metal, regardless of the amount of prior swaging, possessed mechanical properties inferior to as-swaged or stress-relieved metal at room temperature. Although increased swaging produced a considerably finer recrystallized grain size, this was no criterion by which strength or ductility could be predicted for tests at room temperature.

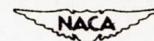
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TABLE I. - SWAGING PROCEDURE OF MOLYBDENUM

Rod diameter, in.	Reduction in area from ingot, percent	Swaging temperature, °F			Remarks
		Lot 1	Lot 2	Lot 3	
1.125 square ingot to 1.125	21.5	2725	2725	2725	a
1.125 to 0.687	70.7	2450	2450	2450	a
0.687 to 0.500	84.5	2275	2275	2275	a
0.500 to 0.300	94.4	1925	1925	1750	b
0.300 to 0.225	96.9	1750	1750	1650	b,c
0.225 to 0.177	98.1	1750	1750	1650	b,c
0.177 to 0.155	98.5	1750	1750	1525	b,c
0.155 to 0.142	98.8	1650	1650	1525	b,c
0.142 to 0.132	98.9	1650	1650	1525	b,c
0.132 to 0.125	99.0	1650	1650	1525	b,c



^a $\pm 175^{\circ}$ F.

^b $\pm 50^{\circ}$ F.

^c ± 0.002 .

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TABLE II. - SHORT-TIME TENSILE STRENGTH, YIELD STRENGTH,
AND DUCTILITY OF MOLYBDENUM

Swaging, percent	Temperature, °F	Ultimate tensile strength, lb/sq in.	Yield strength 0.2 percent offset, lb/sq in.	Elongation in 1.25 inch, percent	Remarks
Lot 1					
99	RT	107,300	90,800	17.2	-
99	RT	104,200	92,100	21.3	-
99	RT	103,500	92,200	23.4	-
99	RT	72,800	-----	0	a
99	1800	38,300	-----	15.6	-
99	1800	37,200	-----	11.3	-
99	1800	36,900	-----	13.8	-
69	RT	99,500	90,100	20.3	-
69	RT	97,100	84,000	17.2	-
69	RT	94,400	-----	21.9	-
69	RT	71,300	-----	0	a
69	1800	39,200	-----	10.9	-
69	1800	37,700	-----	9.4	-
69	1800	37,500	-----	9.0	-
50	RT	96,100	86,400	17.2	-
50	RT	95,200	83,200	16.4	-
50	RT	93,800	87,200	14.1	-
50	RT	49,800	-----	0	a
50	1800	38,400	-----	9.4	-
50	1800	38,000	-----	13.8	-
50	1800	35,800	-----	10.0	-
35	RT	96,700	89,400	15.6	-
35	RT	95,900	89,900	18.4	-
35	RT	91,100	80,600	20.0	-
35	RT	50,900	-----	0	a
35	1800	37,800	-----	10.9	-
35	1800	37,000	-----	10.0	-
35	1800	36,900	-----	12.2	-
Lot 2					
35	RT	84,500	78,600	18.8	-
35	RT	84,000	72,900	25.7	-
35	RT	77,300	67,200	23.5	-
35	RT	70,100	-----	6.3	-
35	RT	74,200	-----	8.6	b
35	RT	86,600	77,500	26.6	c
35	RT	86,000	77,600	31.1	d
35	1800	31,100	-----	16.8	-
35	1800	28,700	-----	21.0	-
22	RT	100,300	95,100	19.5	-
22	RT	99,800	90,200	23.0	-
22	RT	62,300	-----	3.1	b
22	RT	63,500	-----	3.5	c
22	RT	83,700	81,700	28.1	d
22	1800	33,900	-----	13.8	-
22	1800	30,800	-----	7.0	-

^aRecrystallized (1 hr at 2237° F in hydrogen).

^bRecrystallized (taken to 2700° F in vacuum).

^cRecrystallized (1/2 hr at 2275° F + 1/2 hr at 2410° F in hydrogen).

^dStress relieved (1 hr at 1700° F in vacuum).



TABLE II. - Concluded. SHORT-TIME TENSILE STRENGTH, YIELD
STRENGTH AND DUCTILITY OF MOLYBDENUM

Swaging, percent	Temperature, °F	Ultimate tensile strength, lb/sq in.	Yield strength 0.2 percent offset, lb/sq in.	Elongation in 1.25 inch, percent	Remarks
Lot 2 - Concluded					
10	RT	85,400	70,200	34.3	-
10	RT	85,400	76,500	28.1	-
10	RT	85,100	71,500	40.3	-
10	RT	73,200	-----	6.3	b
10	RT	70,200	-----	14.1	c
10	RT	85,100	77,800	29.7	d
10	RT	84,400	73,600	36.7	d
10	1800	28,900	-----	21.8	-
10	1800	25,800	-----	26.4	-
Lot 3 ^e					
99	RT	104,900	94,600	26.6	-
99	RT	102,200	90,100	25.8	-
99	RT	101,800	90,200	25.2	-
99	RT	100,800	88,500	26.6	-
99	RT	99,200	86,500	20.3	-
99	RT	98,700	85,500	26.6	-
99	RT	96,000	81,600	28.9	-
99	RT	78,600	61,400	32.8	f
99	RT	75,400	67,600	29.7	f
99	RT	70,900	63,800	3.1	f
99	RT	96,700	84,900	25.0	g
99	RT	94,100	80,600	28.1	g
99	1800	34,900	-----	8.2	-
99	1800	33,800	-----	11.2	-
99	1800	32,600	-----	10.6	-
50	RT	95,100	83,400	25.8	-
50	RT	94,300	81,000	25.2	-
50	RT	94,000	83,100	21.9	-
50	RT	72,700	44,400	12.7	h
50	RT	71,400	41,700	13.3	h
50	RT	91,800	80,400	28.5	g
50	RT	91,500	83,300	29.7	g
50	1800	33,100	-----	13.3	-
50	1800	32,100	-----	7.5	-
50	1800	29,600	-----	8.7	-
35	RT	91,800	79,600	7.1	-
35	RT	88,700	80,600	8.6	-
35	RT	88,300	76,300	3.1	-
35	RT	70,700	40,700	11.7	h
35	RT	69,900	41,500	11.7	h
35	RT	88,600	80,600	31.3	g
35	RT	86,300	78,600	29.7	g
35	1800	34,900	-----	10.0	-
35	1800	34,600	-----	8.7	-
35	1800	31,500	-----	10.0	-

^bRecrystallized (taken to 2700° F in vacuum).

^cRecrystallized (1/2 hr at 2275° F + 1/2 hr at 2410° F in hydrogen).

^dStress relieved (1 hr at 1700° F in vacuum).

^eData from NACA TN 2915

^fRecrystallized (6 min at 2375° F in hydrogen).

^gStress relieved (1/2 hr at 1742° F in hydrogen).

^hRecrystallized (2 hr at 2642° F in hydrogen).

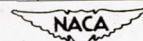
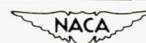
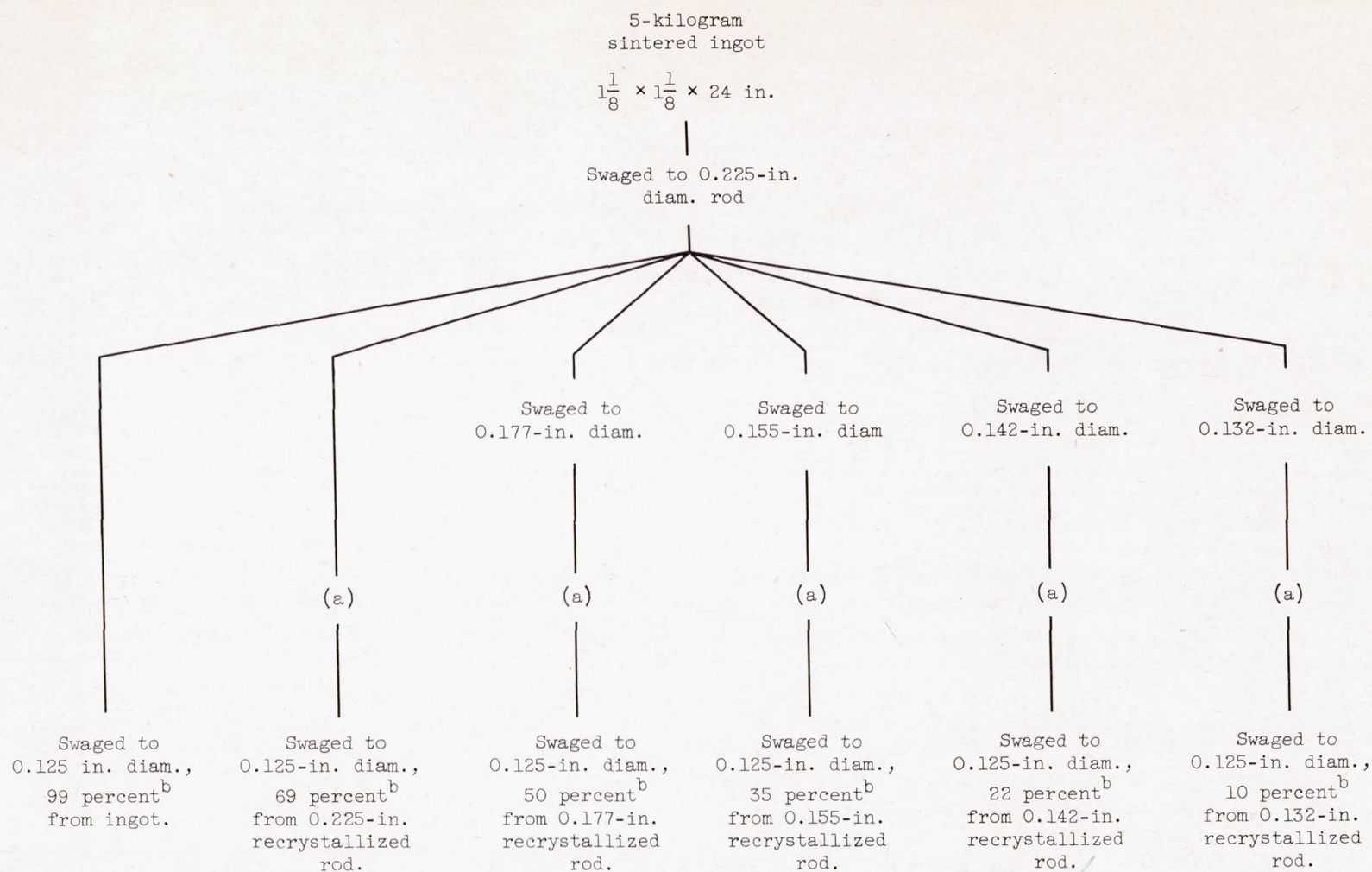


TABLE III. - ANALYSES OF MOLYBDENUM

Element	Lot		
	1	2	3 ^a
Vacuum fusion analysis, percent by weight			
Hydrogen	^b 0.00018	^c 0.00004 ^c 0.00004	^c 0.00002 ^c 0.00002
Nitrogen	^d 0.0007	^d 0.0003 ^d 0.0002	^d 0.0002 ^d 0.0003
Oxygen	^e 0.0025	^d 0.0021 ^d 0.0011	^d 0.0022 ^d 0.0018
Conventional chemical analysis, percent by weight			
Carbon	^f 0.005	^g 0.007 ^g 0.006	^g 0.006 ^g 0.004
Sulphur	^h 0.003	^h 0.003	^h 0.003
Semiquantitative spectrographic analysis, percent by weight. ⁱ			
Boron	0.005	0.005	0.005
Phosphorus	.05	.05	.05
Iron	.01	.01	.01
Magnesium	.05	.05	.05
Lead	.005	.005	.005
Silicon	.05	.05	.05
Bismuth	.01	.01	.01
Chromium	.005	.005	.005
Aluminum	.01	.01	.01
Uranium	.10	.10	.10
Columbium	.05	.05	.05
Vanadium	.05	.05	.05
Copper	.01	.01	.01
Cobalt	.05	.05	.05
Nickel	.01	.01	.01
Manganese	.005	.005	.005
Calcium	.005	.005	.005
Zinc	^j .05	.05	.05
Titanium	^j .05	.05	.05
Antimony	^k 0.005	^k 0.005	^k 0.005
Arsenic	^k 0.005	^k 0.005	^k 0.005
Barium	^k 0.01	^k 0.01	^k 0.01
Lithium	^k 0.005	^k 0.005	^k 0.005
Cadmium	^k 0.01	^k 0.01	^k 0.01
Silver	^k 0.005	^k 0.005	^k 0.005

^aData from NACA TN 2915^bAccuracy, $\pm$ 0.00003^cAccuracy, $\pm$ 0.00002^dAccuracy, $\pm$ 0.0002^eAccuracy, $\pm$ 0.0004^fAccuracy, $\pm$ 0.002^gAccuracy, $\pm$ 0.001^hAccuracy, $\pm$ 0.001ⁱAll percentages by weight are present in less than designated amount.^jSlightly higher than designated amount.^kNot detected (values represent detectable limit).



^aRecrystallized, 6 min. at 2375° F.

^bReduction in cross-sectional area.

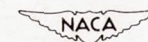
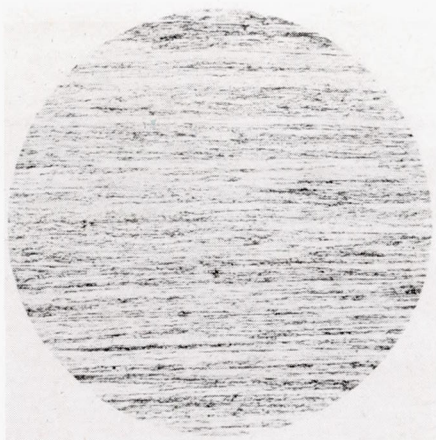
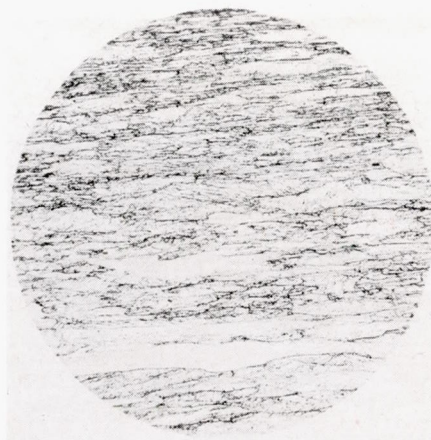


Figure 1. - Processing flow diagram.



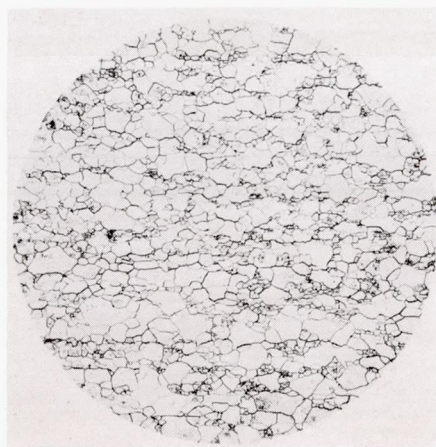
(a) Swaged 99 percent
from ingot.



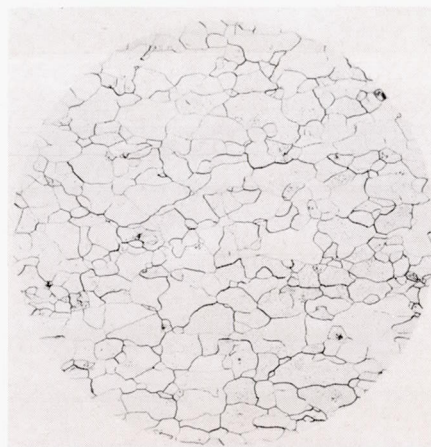
(b) Swaged 69 percent
from 0.225-inch diameter
recrystallized rod.



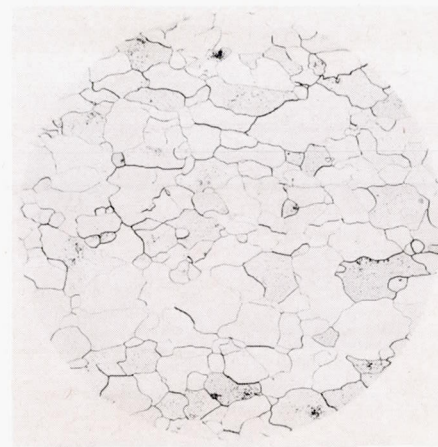
(c) Swaged 50 percent
from 0.177-inch diameter
recrystallized rod.



(a) Recrystallized.



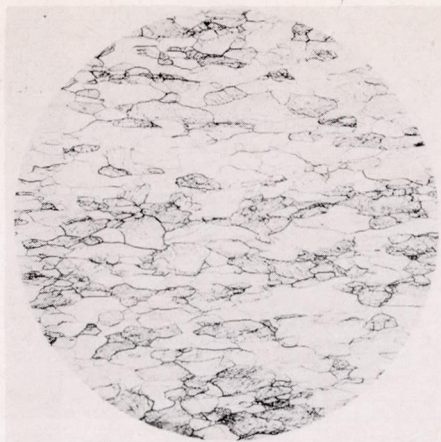
(b) Recrystallized.



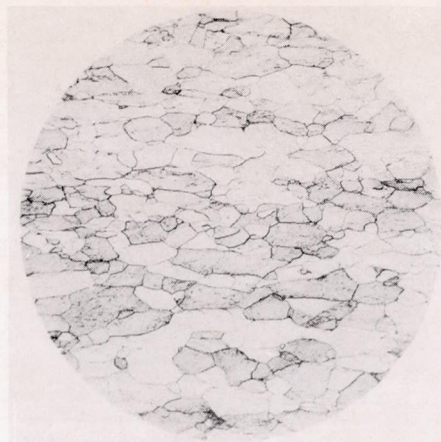
(c) Recrystallized.

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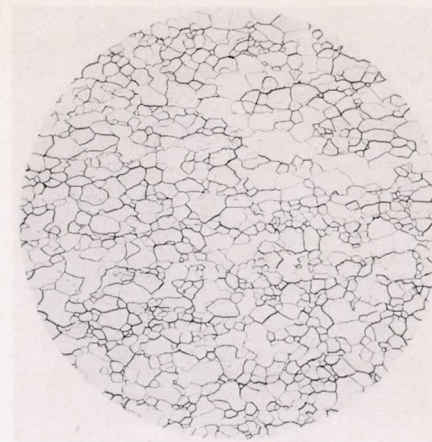
Figure 2. - Representative longitudinal views of 0.125-inch-diameter molybdenum rods swaged various amounts and then recrystallized. Photographed at X100; reduced 36 percent in reproduction.



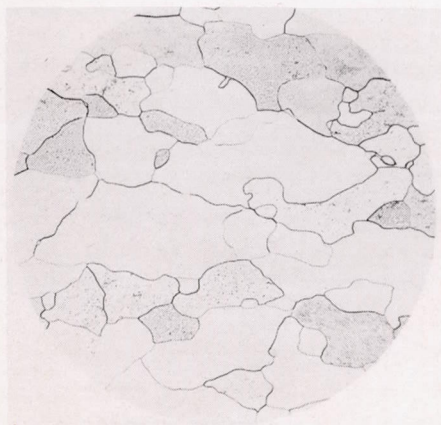
(d) Swaged 35 percent from
0.155-inch diameter
recrystallized rod.



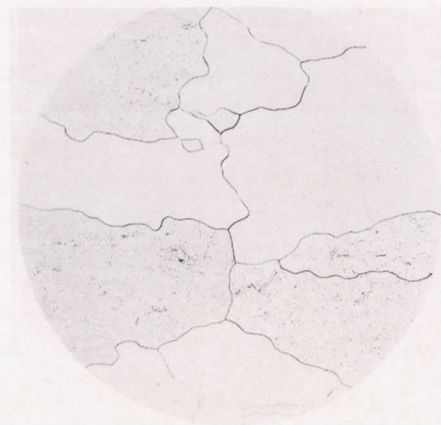
(e) Swaged 22 percent from
0.142-inch diameter
recrystallized rod.



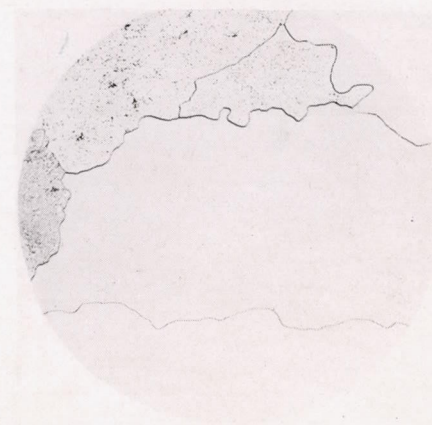
(f) Swaged 10 percent from
0.132-inch diameter
recrystallized rod.



(d) Recrystallized.



(e) Recrystallized.



(f) Recrystallized.

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Figure 2. - Concluded. Representative longitudinal views of 0.125-inch-diameter molybdenum rods swaged various amounts and then recrystallized. Photographed at X100; reduced 36 percent in reproduction.

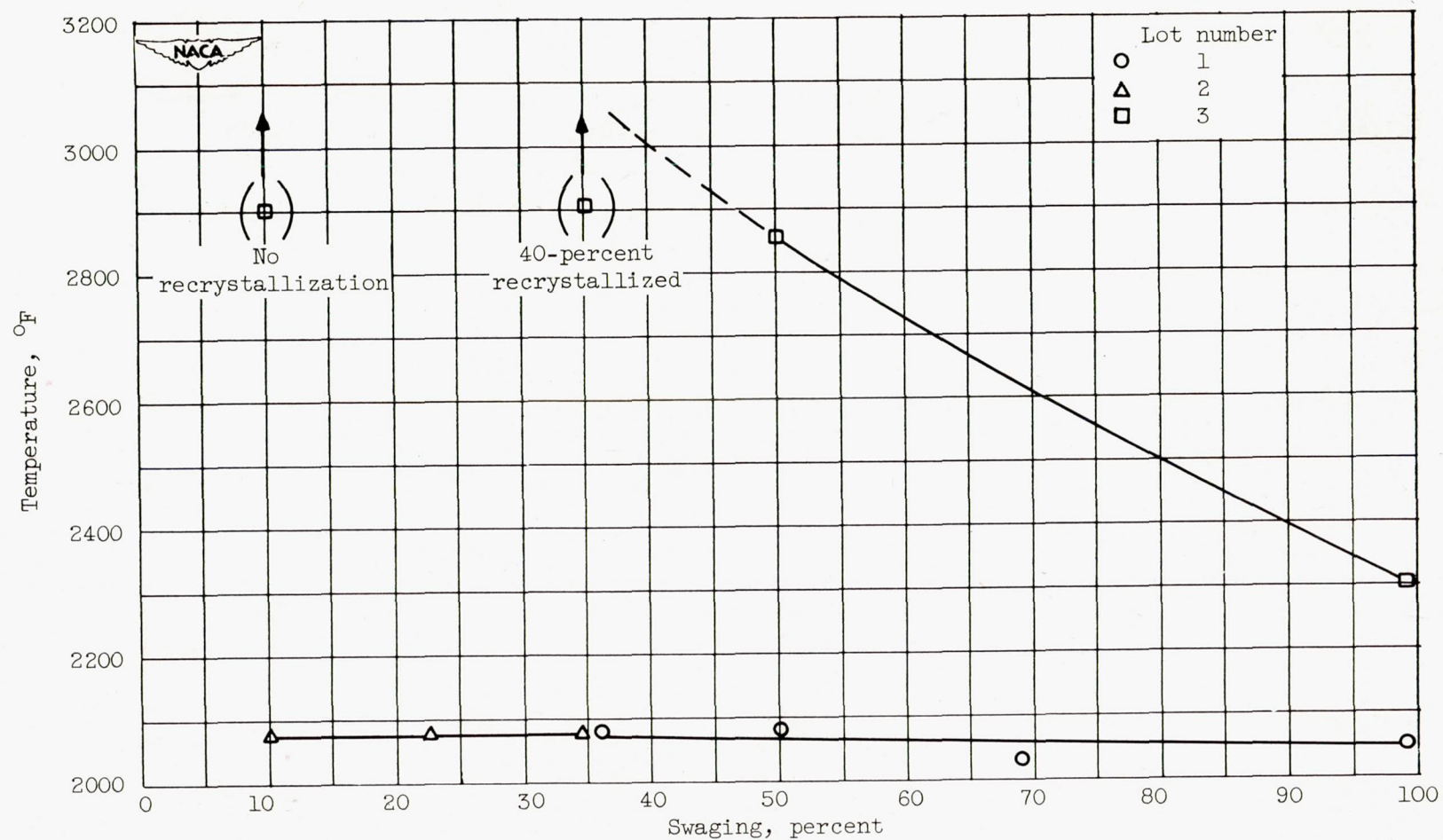
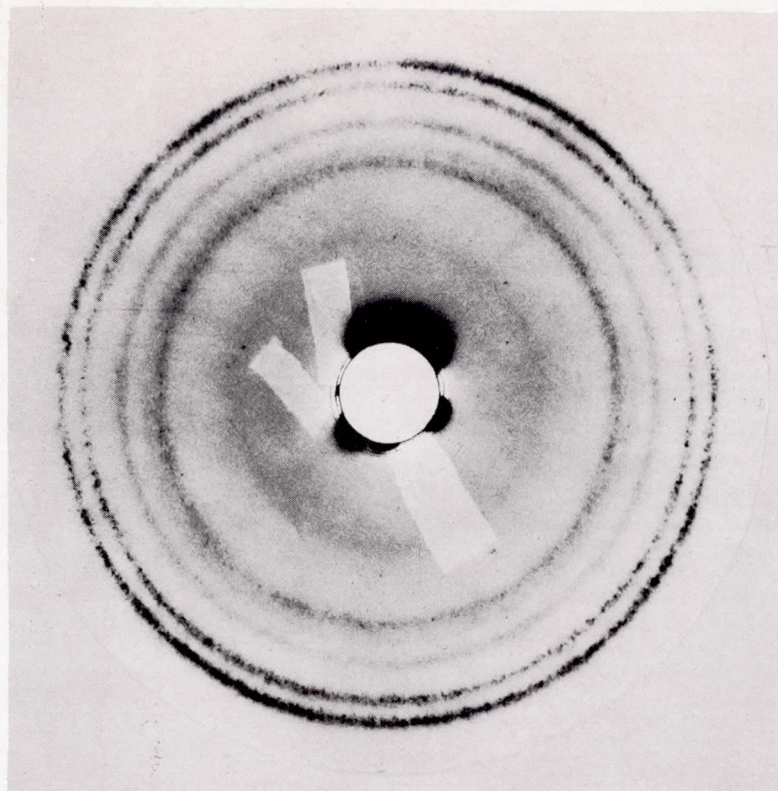
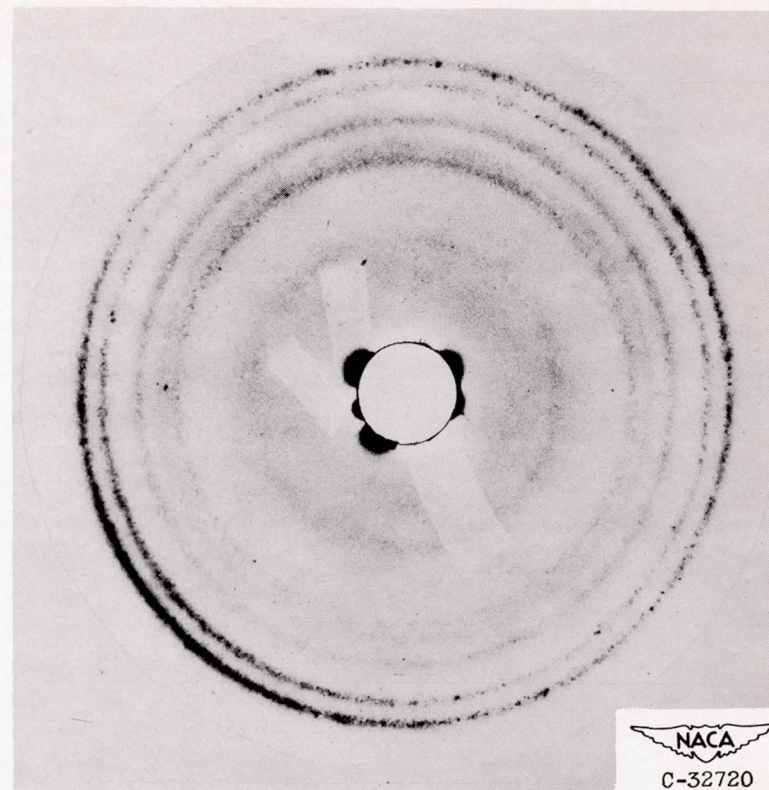


Figure 3. - Effect of swaging on the 1-hour total recrystallization temperature of molybdenum in argon.



(a) Swaged 99 percent.



(b) Swaged 35 percent.

Figure 4. - Back reflection photograms of lot 3 molybdenum. Copper radiation; 46-hour exposure.

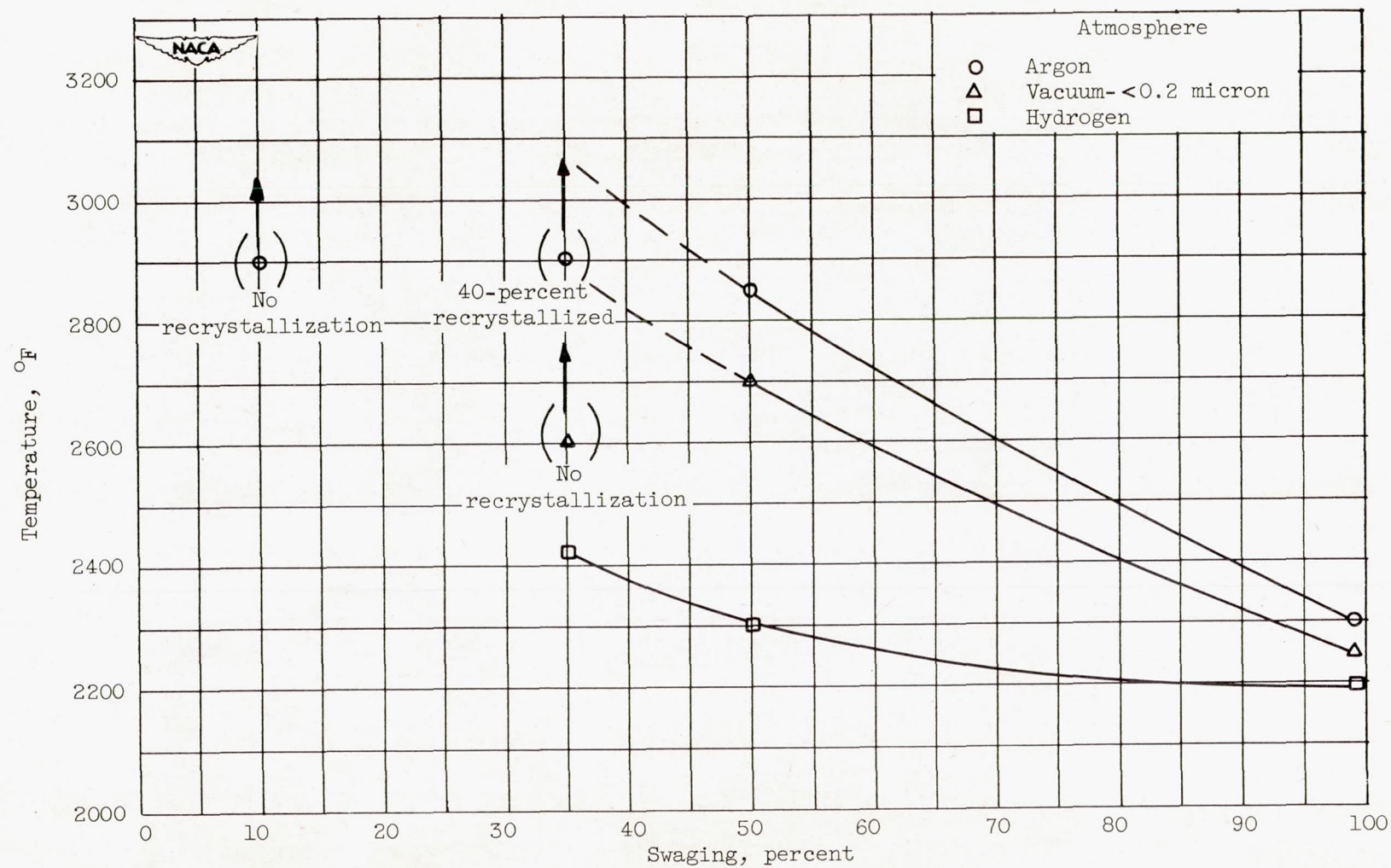
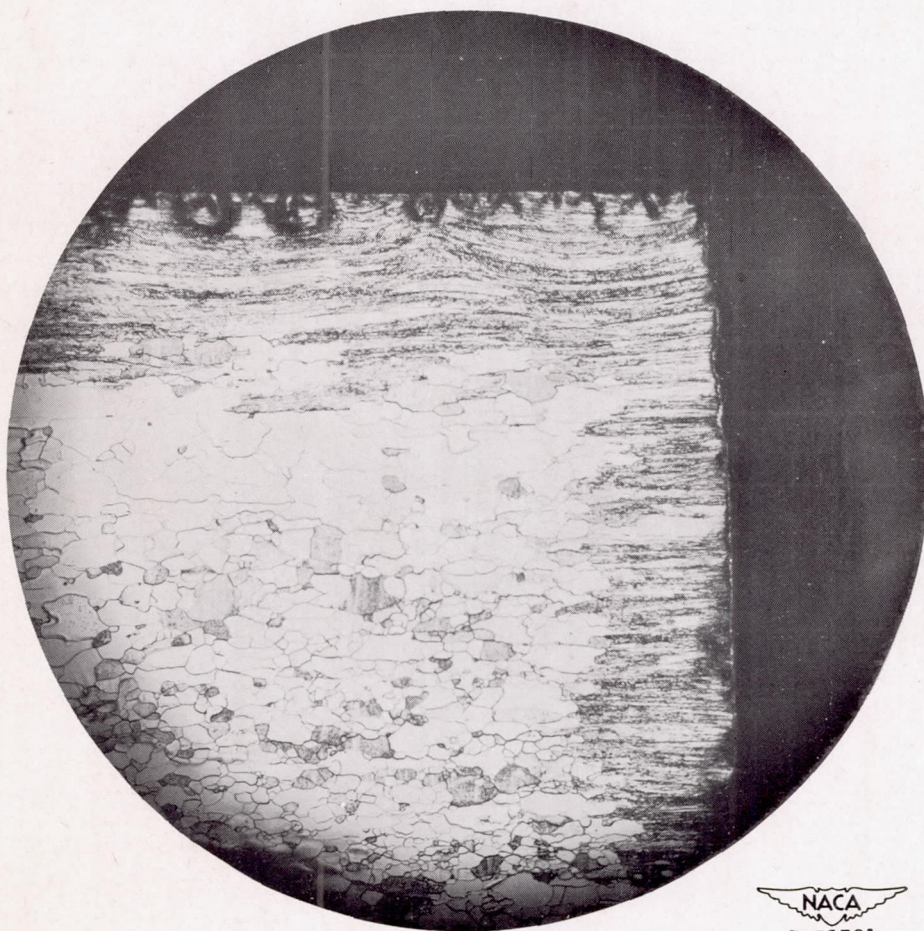


Figure 5. - Effect of atmosphere on total 1-hour recrystallization temperature of molybdenum (lot 3).



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Figure 6. - Longitudinal view of unrecrystallized surface grains. X50.

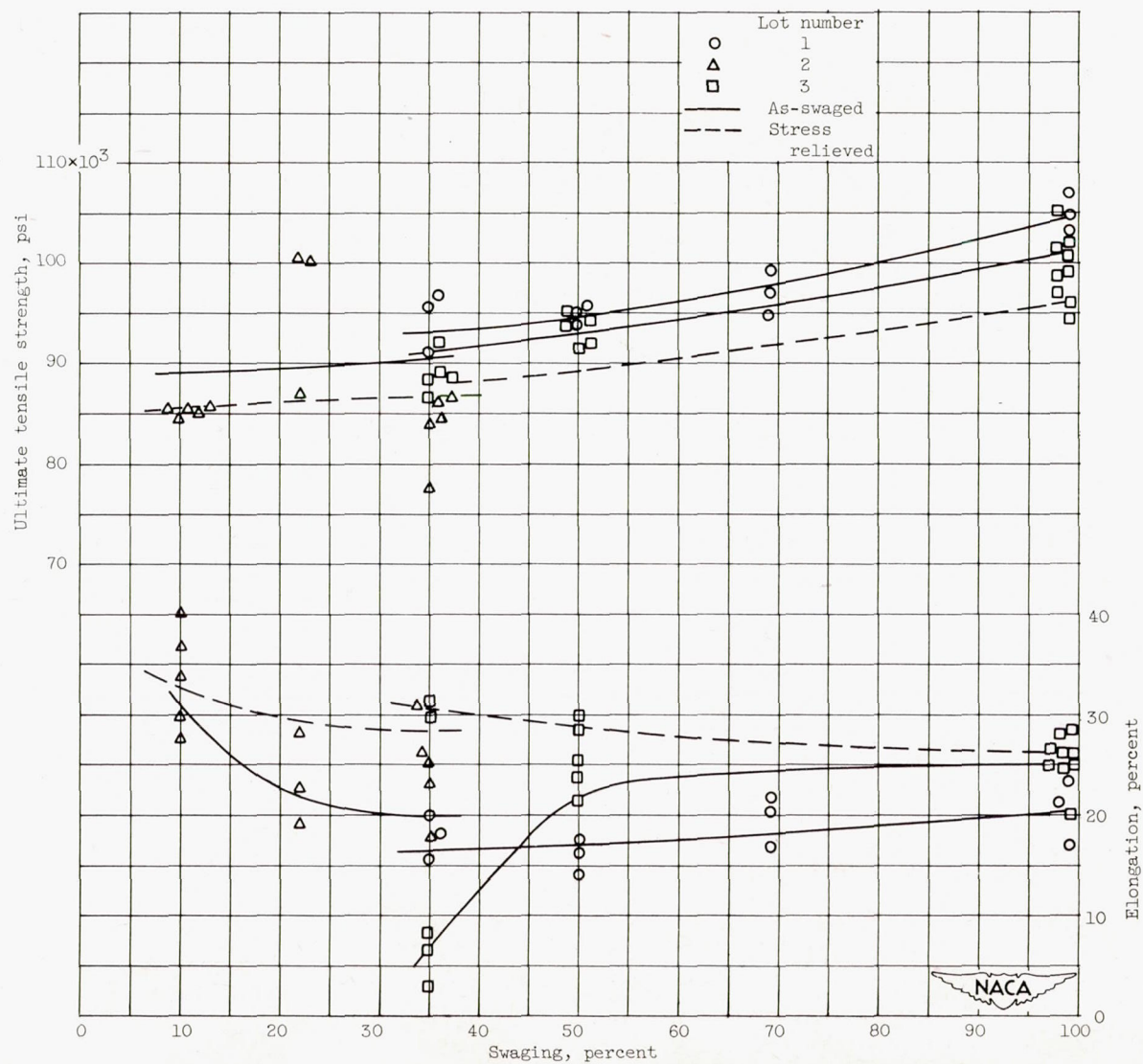


Figure 7. - Effect of swaging on strength and ductility of molybdenum at room temperature.

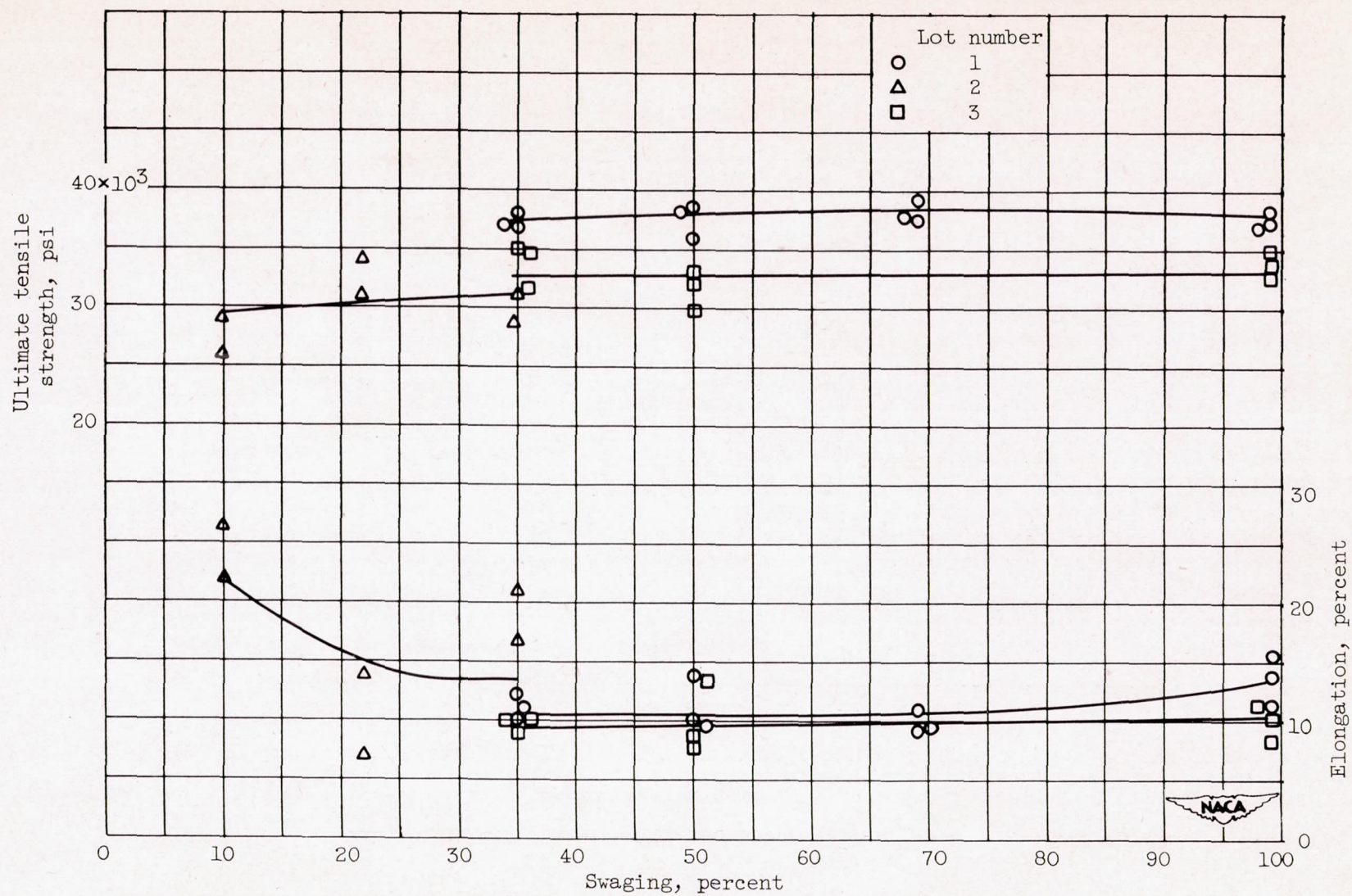


Figure 8. - Effect of swaging on strength and ductility of molybdenum at 1800° F.

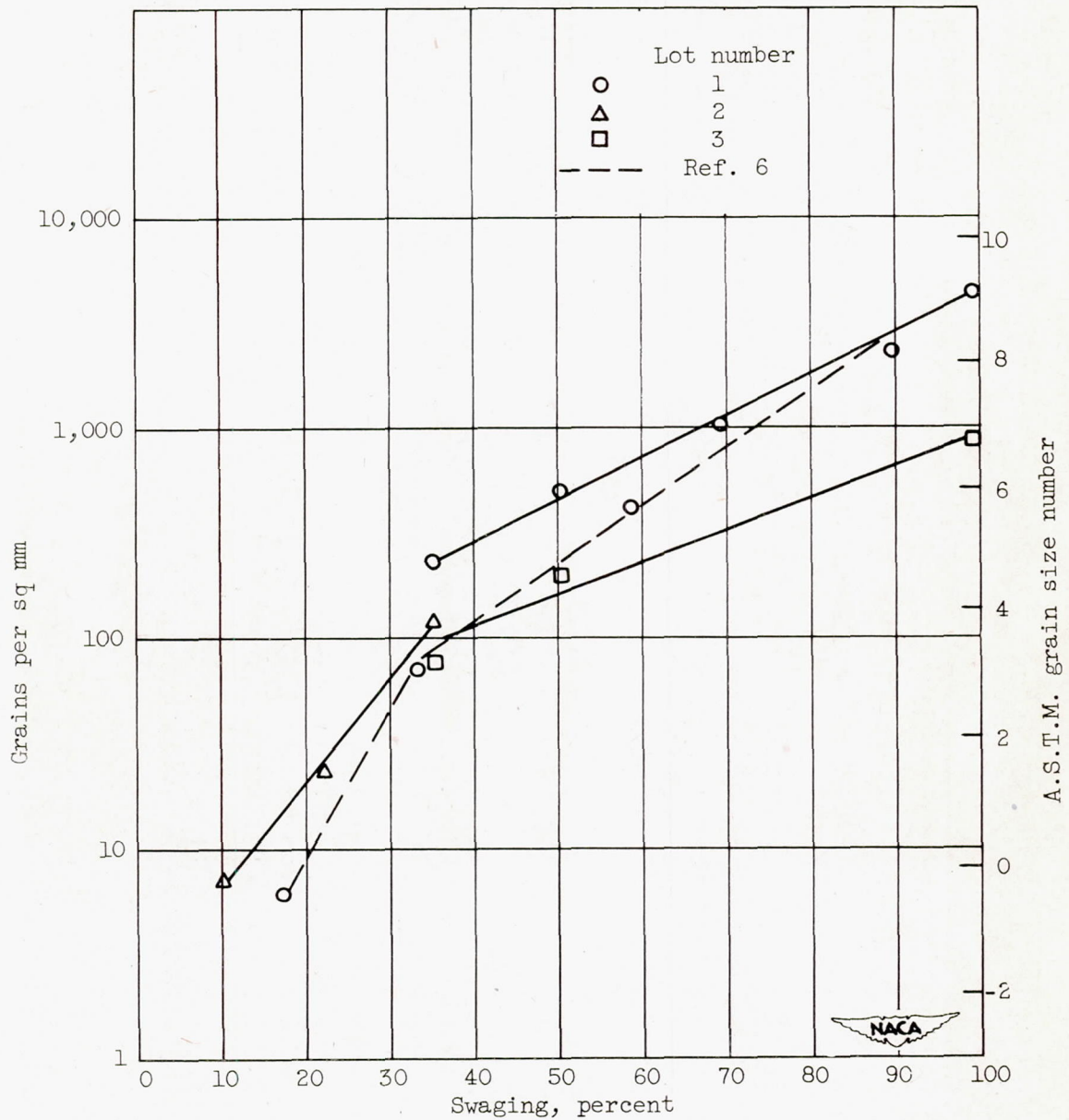


Figure 9. - Effect of prior swaging on grain size of recrystallized molybdenum.

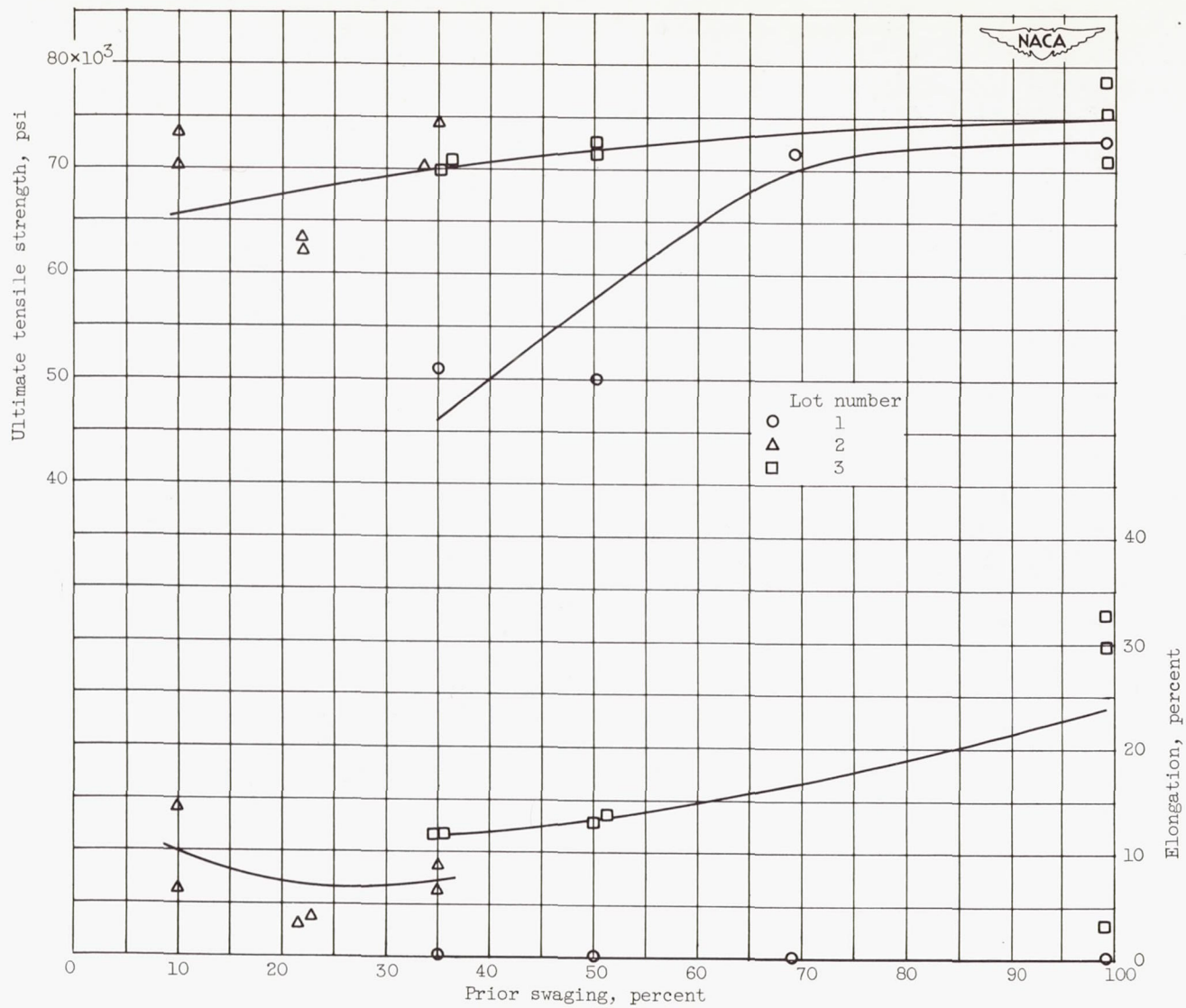


Figure 10. - Effect of prior swaging on room-temperature strength and ductility of recrystallized molybdenum.